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## MONITORING LIQUID PROPELLANT DURING SURVEILLANCE

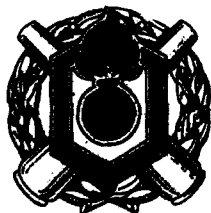
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Armament Engineering Directorate

Picatinny Arsenal, New Jersey

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methods selected were tested in-depth, and comparison studies were conducted with several. As a result, a capability is available to monitor liquid propellants during storage, to establish kinetics and decomposition mechanisms, to establish specifications, and to permit evaluations of additives. Data are being generated from ongoing studies to permit predictions of long-term effects such as pressure buildup, effect of contaminants, and shelf-life.

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## INTRODUCTION

The advantageous features of liquid propellants in the areas of logistics, ballistics, cost savings, and its being categorized as an insensitive munition have prompted an extensive surveillance program. The commitment to develop insensitive munitions systems has led to the testing of liquid gun propellants as a potential replacement of solid propellants used in the 105-mm and 155-mm guns.

The integrity, stability, and safety of these propellants, after being stored for extended periods of time, is necessary in order to meet ballistic requirements. As part of this program, there was a need to provide techniques to monitor and characterize the liquid propellants during both accelerated and long-term storage. The use of the liquid propellants in diverse gun applications necessitates the ability for their characterization which has resulted in the development of analytical monitoring capabilities. These techniques will provide the ability to monitor the composition and integrity of liquid propellants during storage. At present, fail/safe criteria are not available for liquid propellants under long-term storage conditions, and a methodology is being developed to establish these criteria. To establish these criteria, studies are being conducted to determine aging effect on the stability and storability of liquid propellants. The rate data generated in the accelerated testing are being used for the determination of the various fail/safe criteria, and the long-term storage testing will establish shelf life for liquid propellants.

## BACKGROUND

The liquid propellant (LP) system under investigation is a stoichiometric mixture of 61% hydroxylammonium nitrate (HAN) and 19% triethanolammoniumnitrate (TEAN) in 20% water. This mixture is stoichiometric with a molar ratio of 7:1 for conversion to carbon dioxide, water, and nitrogen. This liquid propellant composition designated LP 1846 is the prime candidate currently under consideration.

Determining the effects of temperature, inhibitors, and contaminants on the stability of liquid propellants as well as providing analytical techniques for monitoring liquid propellant before and after exposure are the primary goals of this investigation. The analyses are required for identification and quantitation of major propellant components, contaminants, and degradation products which will provide a basis for establishing the various criteria. The criteria of interest include decomposition kinetics and mechanisms, propellant composition and ballistics relationships, recommendations for propellant specifications, pressure buildup during storage, propellant shelf-life, and safety factors. Low level concentrations of contaminants or impurities have been identified previously (refs 1 through 3) such as ammonium nitrate (AN), nitric acid, NOx's ( $N_2O$  and  $NO_2$ ), nitrogen, and trace amounts of transition metal ions. This list is not all inclusive and may be changed during this investigation.

## PROCEDURE

A review of techniques as well as investigations to confirm reliability and to optimize several of the techniques was presented in previous reports (refs 1 through 3). A comparison chart of analytical methods is shown in table 1. This chart will be completed after comparisons are made through intra-lab studies.

Nitric acid is determined using a non-aqueous titrimetric method on a Metrohm model E536 Potentiograph and a Model 655 Dosimat.

HAN, TEAN, and AN are determined by ion chromatography (IC) using a Waters system.

The gaseous decomposition products ( $\text{NO}_x$  and  $\text{N}_2$ ) are determined by gas chromatography.

Metals analyses were compared on several systems including polarography, atomic absorption spectroscopy (AA), graphite furnace atomic absorption spectrophotometry (GPAA), inductively coupled plasma spectrophotometry (ICP), inductively coupled plasma spectrophotometry/mass spectrometry (ICP/MS) and ion chromatography (refs 1 through 3). Of the above, GPAA appears to be the best choice as a reference method but not as a routine technique since it is very time consuming. The data in this report were obtained on a Waters' IC which was selected for its ability to detect at minimum levels required for this study as well as speed and ability to differentiate valence states. Optimization studies are still underway on the IC.

LP samples are currently undergoing accelerated and long-term storage testing over a range of temperatures and levels of contaminants and inhibitors. The accelerated experiments are being conducted with small scale apparatus which has been discussed previously (refs 1 and 2). The long-term tests are being conducted on a larger scale in order to provide sufficient sample for analytics, accelerating rate calorimetry (ARC), and ballistics.

## RESULTS AND DISCUSSION

Several of the analyses are very critical to this program since these monitor the levels of contaminants most detrimental to the stability of LP. These are the determinations of both free nitric acid and transition metals. An in-depth investigation of the use of titrimetry for the determination of nitric acid using a nonaqueous technique was selected and discussed in previous reports (refs 1 through 3). The titration of HAN and TEAN by aqueous and nonaqueous methods have proven to be less than desirable. As a result, ion chromatography has been investigated and found to provide not only HAN



and TEAN but AN, ethanolammonium nitrate (EAN), and diethanolammonium nitrate (DEAN) in one chromatogram. The IC method has been optimized and found to be not only precise but accurate. For the transition metals, IC was selected for its sensitivity at the trace levels necessary for this program. IC has the capability to detect tenths of a part per million (ppm) in the LP matrix. Its speed and ability to differentiate valence states of numerous metals in one determination is also advantageous. The ability to determine valence states will be useful to understanding the decomposition mechanism due to metal catalysis.

The analysis of HAN and TEAN were optimized using ion chromatography with a modified integration algorithm. This method is capable of detecting not only HAN and TEAN but also AN, EAN, and DEAN. An analysis of a synthetic standard of hydrochloride salts of HAN and TEAN is presented in table 2. A comparison of the data previously obtained by titration for TEAN illustrates the value of IC since it is not effected by other weak acids as was the titration methods. It was also apparent in the titration of HAN by the nonaqueous and even more so by the aqueous that the values were always on the low side. This was probably due to some type of equilibrium or inability to titrate all of the HAN present.

The transition metals (especially iron, copper, nickel, and chromium) and aluminum are known to accelerate the decomposition of HAN-based propellants. Considerable effort has been expended in this area to provide a reliable technique. Analytical methods which have been considered include: polarography, ion chromatography, inductively coupled plasma, atomic absorption, graphite furnace AA, and inductively coupled plasma/mass spectrometry.

Initially, polarography was used to develop methods for the transition metals (refs 2 and 3). The ability of the liquid propellant to act as oxidizer, reducing agent, and/or chelating agent for species of the transition metals complicated the polarographic analyses. This was made more evident when ICP data were compared to polarographic data for iron (table 3). The difference between the two methods illustrates that most of the  $\text{Fe}^{+3}$  in the liquid propellant is in a complexed state and not available for polarographic detection as  $\text{Fe}^{+3}$  (refs 2 and 3).

Analyses of liquid propellant samples were conducted by both the inductively coupled plasma spectrometer and atomic absorption spectroscopy. Results from several laboratories indicated that ICP analysis of metals in liquid propellants is very matrix sensitive and not at all straight-forward. The levels reported on the same sample could vary as much as 2 to 10 times what was actually present. A comparison was made using ICP with internal and external standardization with graphite furnace AA as reference (ref 3). The data from this study is shown in table 4. Careful use of the ICP

with internal standardization will produce reliable results. The internal standard was found necessary to correct for plasma and sample viscosity fluctuations due to matrix effects. The use of AA without a graphite furnace was insensitive to the levels necessary for this program.

As a result of the difficulties encountered with polarography and other techniques, IC was investigated and feasibility studies conducted on metal spiked samples of liquid propellant. The data indicate that IC will not only provide quantitative data for all species of interest but also the oxidation state of the metal. This information will be useful in determining the role of metal impurities in liquid propellants.

For the ion chromatography of the +2 transition metals and  $\text{Fe}^{+3}$ , a  $\mu$ Bondapak C18 column is used for the separation. The eluant is 2mM NaOS/15-35mM tartaric acid/5% acetonitrile (pH adjusted to 3.65 with 50% NaOH) at a flow rate of 1.0 ml/min. The post column reactant (PAR) flow rate was 0.5 ml/min. An ultraviolet (UV) detector at 520 nm was required for the identification of the metals present.

Aluminum and chromium are separated on the same type of column but with different eluants, post column reactants and UV wavelengths. For aluminum, the eluant is 0.1 M sulfuric acid/0.2 M ammonium sulfate at a flow rate of 0.9 ml/min. The post column reactant is tiron at a flow rate of 0.5 ml/min with UV detection at 310 nm. The eluant for chromium (chromate) is 5 mM tetrabutyl ammonium phosphate at a flow rate of 1.0 ml/min using the UV detector at 365 nm.

The LP samples were digested in concentrated nitric acid. The digestion is necessary to eliminate the possibility of metal chelation by the liquid propellant ingredients, namely HAN and TEAN. After the HAN and TEAN are destroyed, a dilute acid solution is added to the residue to provide a 1/10 dilution of the original sample. It is necessary for the solution to be acidic to prevent precipitation of metal hydroxides. The sample injection volume was 100  $\mu$ l. Chromatograms of the +2 transition metal standards plus  $\text{Fe}^{+3}$  are shown in figures 1 through 4. All of these metals ( $\text{Fe}^{+3}$ ,  $\text{Cu}^{+2}$ ,  $\text{Ni}^{+2}$ , and  $\text{Fe}^{+2}$ ) can be obtained in one chromatographic separation. The  $\text{Fe}^{+3}$  elutes close to the void volume and can be difficult to reproduce accurately when using strong eluant (50 mM tartaric acid). Therefore, if necessary, the tartaric acid concentration is lowered to 35 mM resulting in the  $\text{Fe}^{+3}$  eluting at a later retention time. A chromatogram of chromium (chromate) standard is shown in figure 5 and an aluminum standard in figure 6. Spiking of the LP sample was used to verify the peak identity in the LP samples. A chromatogram of LP 1846 after digestion and dilution in 25 ml of nitric acid acidified (pH = 1) distilled water is shown in figure 7. The peaks which were detected for this sample are shown in the area tabulations. The levels of transition metals detected for this sample were 0.70 ppm  $\text{Fe}^{+3}$  and 0.03 ppm  $\text{Cu}^{+2}$  which is in good agreement with the data in table 5 which compares this sample and others by GPAA and ICP.

With the advent of more reliable analytical techniques, correlations of recent accelerated stability tests have been more in line with previously reported LP 1846 stability data. A tabulation of rate data for completed and on-going accelerated pressure-time studies are presented in table 6. The data indicate that in the on-going tests at 30°C and 50°C there is no significant pressure rise. Some of the samples at each of the temperatures were spiked with nitric acid and ion ( $\text{Fe}^{+3}$ ). Nitric acid was chosen since it was found to have a strong catalytic effect on LP decomposition. Iron ( $\text{Fe}^{+3}$ ) was chosen since it is always regenerated after reaction with LP to its catalytic state. The reaction of  $\text{Fe}^{+3}$  with LP results in its reduction to  $\text{Fe}^{+2}$ . It is reoxidized back to  $\text{Fe}^{+3}$  by the nitric acid formed in the original reaction. This cycle continues until all of the HAN and then TEAN is consumed. It was noted previously that in the analytical data some of the completed tests showed some degradation at 50°C (table 7). These samples contained higher levels of metal contamination than those in the on-going tests. In these lower temperature levels and at 65°C with no contaminants, the pressure is not as good an indicator of propellant degradation as is the analytical data. Whereas, at 65°C the contaminated samples in the completed and on-going tests all showed pressure buildup when the contaminant levels were  $>0.1\%$  nitric acid and  $>1$  ppm of metal ( $\text{Fe}^{+3}$ ). These samples produced significant pressure buildup and therefore unacceptable rates of decomposition (table 6). The inhibitor study at 65°C after 6 months reveals a considerable reduction in sample degradation when high levels of metals ( $\text{Fe}^{+3}$ ) are present (table 6). The rate has been reduced to less than 70% for 4 ppm  $\text{Fe}^{+3}$  when the inhibitor is present. The samples containing only inhibitor are showing comparable results to the control samples. The analyses of the on-going samples after completion of the study will be used to complement the rate data. Long-term storage of larger lots of LP are currently underway at ambient, 50°C, and 65°C in polyethylene containers. After completion of calibration of a temperature cycling chamber, this test will also be conducted over the temperature range of ambient to 65°C. Samples from the long-term tests will be used to obtain analytical, ballistic, and accelerating rate calorimetry (ARC) correlation data. The combination of the accelerated and long-term testing will be used to establish and/or recommend storage, contaminant effects, safety, ballistic, handling, specification, mechanistic, kinetic, and potential flags criteria for LP.

## SUMMARY

The analytical techniques used for this program complement the rate and storage data being generated. These methods have been thoroughly investigated to provide the needs of these studies.

The accelerated test data have shown the effects of contaminants at elevated temperatures and verify the need for strict specifications. The use of inhibitors were observed to have a strong effect on lowering the rate of decomposition and will need to be studied further at other levels and types of transition metal contamination. In subsequent reports, the on-going accelerated and long-term tests data will be used to supplement these data and further these conclusions.

Table 1. LP component analyses chart

Component Contaminant	Cost \$K	Sample Size, g	Accuracy %	Precision +/-	Rapidity Time/#	Complexity (10=high)	Toxicity (10=high)	MDL %/ppm
<b>A-HAN</b>								
1-Tit/Aq	8	0.5	-3.8	0.1	20min (A, B)	3	5	NA
2-Tit/NonAq	8	0.5	-1.0	0.1	20min (A, B, D)	3	5	NA
3-IonChrom	35	0.04	0.9	0.3	20min (A, B, D)	4	2	NA
4-FTIR	50-75	-	-	-	10min (A, B, C, D, E)	6	-	NA
<b>B-TEAN</b>								
1-Tit/Aq	8	0.5	10.0	0.8	20min (A, B)	3	5	NA
2-Tit/NonAq	8	0.5	10.0	0.8	20min (A, B, D)	3	5	NA
3-IonChrom	35	0.04	3.8	0.8	20min (A, B, D)	4	2	NA
4-FTIR	50-75	-	-	-	10min (A, B, C, D, E)	6	-	NA
<b>C-Water</b>								
1-Tit/KF	3	0.5	0.5	0.1	10min	2	3	0.05
2-FTIR	50-75	-	-	-	10min (A, B, C, D, E)	6	-	-
<b>D-AN</b>								
1-Tit/NonAq	8	0.5	NA	1.0	10min (A, B, C, D, E)	3	5	0.1
2-IonChrom	35	0.04	0.5	0.5	20min (A, B, D)	4	2	0.02
3-FTIR	50-75	-	-	-	10min (A, B, C, D, E)	6	-	-
<b>E-Nitric Acid</b>								
1-Tit-Aq	8	0.5	5.0	1.0	10min	2	3	0.01%
2-Tit-Aq	8	28.0	19.6	5.0	10min	2	3	NA
3-Tit-NonAq	8	0.5	3.5	0.2	10min	2	3	0.01%
4-FTIR	50-75	-	-	-	10min (A, B, C, D, E)	6	-	-
<b>F-Metals</b>								
1-ICP/ES	55-100	1/9*	25-55	1.0	20min	6	2	<0.1ppm
2-ICP/IS	55-100	0/0	5.9	1.0	20min	6	2	<0.1ppm
3-ICP/MS	120	1/100*	5-25	1.0	20min	7	2	<0.1ppm
4-GPAA	85	1/10*	1.0	2.0	120min (Ref/Slow)	5	2	<0.01ppm
5-AA	50	1/10*	NA	NA	120min (Slow/Insens)	4	2	10ppm (NA)
6-IonChrom	30	3.0	5.0	2.0	30min	4	2	<0.1ppm

NOTE: \*=dilution

NA=not applicable

Table 2. Comparison of aqueous and nonaqueous synthetic LP 1846  
HACL/TEACL titrations

Titration Type	Diluent	Percent Acetone	Reaction Time, min	Titrant Volume, mls	
				HA•Cl	TEA•Cl
Non-Aq	Ethanol	1.0%	0.0	14.91	17.28
				<u>14.87</u>	<u>17.23</u>
				±0.02	±0.025
		5.0%	0.0	14.95	17.33
				<u>14.93</u>	<u>17.31</u>
				±0.01	±0.01
		10.0%	0.0	14.93	17.32
				<u>14.87</u>	<u>17.26</u>
				±0.03	±0.03
		<u>10.0%</u>	<u>15.0</u>	<u>14.89</u>	<u>17.26</u>
		Actual Concentration		60.8% HAN	19.2% TEAN
		Experimental		61.4% HAN	21.6% TEAN
Aqueous	Water	1.0%	0.0	17.19	20.13
				<u>17.14</u>	<u>20.06</u>
				±0.025	±0.035
		5.0%	0.0	17.11	20.03
				<u>17.20</u>	<u>20.10</u>
				±0.045	±0.035
		10.0%	0.0	17.14	20.03
				<u>17.15</u>	<u>20.02</u>
				±0.005	±0.005
		<u>10.0%</u>	<u>15.0</u>	<u>17.14</u>	<u>20.04</u>
		Actual Concentration		60.8% HAN	19.2% TEAN
		Experimental		58.5% HAN	21.9% TEAN

Note: High purity hydroxylammonium hydrochloride and triethanolammonium hydrochloride was used to prepare solutions containing the same amount of cations (hydroxylamine or triethanolamine) which would be present in the nitrate solutions of LP 1846.

Table 3. Metals analysis of two lots of LP 1846 by ICP

<u>Metal</u>	<u>LP-2, ppm</u>	<u>LP-3, ppm</u>
Iron	<0.09	2.06 (polarography:0.31ppmFe <sup>+3</sup> )
Chromium	0.74	0.40
Copper	<0.18	<0.17
Nickel	0.88	0.34
Cobalt	<0.09	<0.09
Lead	<0.87	<0.87
Tin	3.06	3.03

Note: The difference between polarography and ICP/ES illustrates that the Fe<sup>+3</sup> in the LP is complexed and not available as Fe<sup>+3</sup>.

Table 4. Comparison of graphite furnace AA and ICP metals analyses of LP 1845 lot 1845-01-02

<u>Metal</u>	<u>ICP (ES)/(IS) and GPAA</u>	<u>GPAA Det Limit</u>	<u>ICP Det Limit</u>
Al	<0.075 (ES#2)	0.04 ppb	12 ppb
	<0.020 (ES#2)	0.02 ppb	8 ppb
Cu	0.11 ppm (ES#1)		
Cu	0.24 ppm (IS)		
	<0.0100 (ES#2)	0.01 ppb	5 ppb
Cr	0.02 ppm (ES#1)		
Cr	0.03 ppb (IS)		
	<0.020 (ES#2)	0.10 ppb	20 ppb
Ni	0.00 ppm (ES#1)		
Ni	0.04 ppm (IS)		
	1.0 (ES#2)	0.02 ppb	16 ppb
Fe	0.87 ppm (ES#1)		
Fe	1.76 ppm (IS)		
Fe	1.87 ppm (GPAA)		

Note:

IS = internal standard technique. Scandium used as IS.

ES = external standard technique (no internal standard).

GPAA = graphite furnace AA.

Dilution: GPAA=1/100, ICP #1=0, ICP #2=1/9th.

Same results for Fe<sup>+3</sup> in Lot 292: 0.16 ppm (IS) / <0.01 ppm (ES).

Table 5. Liquid propellant lot analyses

<u>Component</u>	<u>LP-1</u> <u>LP1846</u>		<u>LP1846</u> <u>ABY87F</u> <u>S2C013</u> <u>(LP-2)</u>		<u>LP-3</u> <u>1846-01</u> <u>292</u> <u>(11/87)</u>		<u>LP1845</u> <u>LP1845-01</u> <u>Cont-2</u>		<u>LP1846-03</u> <u>Cont-Orig</u>		<u>LP1846-03</u> <u>Cont-8</u>		<u>LP1846-03</u> <u>Cont-11</u> <u>(LP-4)</u>	
% HAN	60.41		59.29		59.40		63.47		61.97		61.46		61.23	
GEO(Tit)														
GEO(IC)														
BRL					60.09		62.57		61.0♥		60.99*		60.85*	
BRL					60.07*									
% TEAN														
GEO(Tit)	20.11*		19.27		20.34		22.82*		21.28*		21.16*		20.94	
GEO(IC)														
BRL					19.79		20.50		19.6♥		19.40*		19.46*	
BRL					21.23*									
% Water														
GEO	18.60		19.72		19.49		16.46		15.93 }16.26		19.48		19.42 }19.58	
BRL					19.80				19.7♥				(19.75)} 20.18*	
% Nitric acid														
GEO	0.84		0.44		0.033		0.31		0.04 }0.06		0.07		0.08 }0.07	
BRL									(0.07)} 0.07				(0.05)} 0.027*	
BRL					0.015		0.29		0.005		0.004*			
BRL					0.155*									
% AN														
GEO(Tit)	0.71		0.89		0.51								0.80	
GEO(IC)			0.36		0.48		[0.36]						[0.22]	

NOTE: \*Not corrected for (AN). () = Data 7/89. [] = Data 8/89. ♥-Thiokol \* = Data 10/6/89. } = Avg data.



Table 5. (cont)

ppm Metals	LP-1 <u>LP1846</u>	LP1846 ABY87F <u>S2C013</u> (LP-2)	LP-3 <u>1846-01</u>	LP1845 <u>292</u>	LP1845-01 <u>Cont-2</u>	LP1846-03 <u>Cont-Orig</u>	LP1846-03 <u>Cont-8</u>	LP1846-03 <u>Cont-11</u>
Aluminum								
ICP/ES #2			<0.25	<0.075	<0.075			
ICP/MS					0.113			0.084
Chromium								
ICP/IS					0.03			
ICP/ES #1					0.02			
ICP/ES #2		0.736	0.343	1.0	<0.010			
ICP/MS					0.162			0.202
Copper								
ICP/IS					0.24			
ICP/ES #1					0.11			
ICP/ES #2		<0.175	<0.174	<0.020	<0.020			
ICP/MS					0.124			0.005
Iron								
GPA					1.87			
ICP/IS				0.16	1.76			
ICP/ES #1				<0.01	0.87			
ICP/ES #2		<0.088	2.06	<0.010	1.0			
ICP/MS					1.450			0.720
Nickel								
ICP/IS					0.04			
ICP/ES #1					0.00			
ICP/ES #2		0.879	0.394	1.0	<0.020			
ICP/MS					0.029			0.049

Note: ICP=inductively coupled plasma; IS=internal standard(Sc); ES=external std(1=no dilution;2=1/9 dil); MS=mass spectrometer; GPA=graphite furnace AA; IC=ion chromatography.

Table 6. Rate data for pressure time studies with LP 1846

<u>Sample</u>	<u>Temperature(°C)</u>	<u>Rate @20%Ullage mm/Hg/day</u>
0.7-4 ppmFe	30	0
0.1-0.3% acid	30	0
0.7-4 ppm Fe	50	0
0.1-0.3% acid	50	0
0.7 ppm Fe	65	0
1.5 ppm Fe	65	3.8
2.0 ppm Fe	65	14.3
4 ppm Fe	65	17.1
5 ppm Fe	65	35.8
0.1% acid	65	0
0.2% acid	65	4.0
0.3% acid	65	9.0
0.5% acid	65	22.4
1.0% acid	65	43.6
Inhibitor (Deq)	65	0
Deq/4 ppm Fe	65	11.9

NOTE: Inhibitor being used is a phosphonate type called Dequest 2061.

Table 7. Composition analysis of LP 1846 before and after exposure and rate of decomposition as a function of temperature and contaminants at 65% ullage

LP 1846	Temp, °C	%HAN	%TEAN	%Water	%AN	%HNO <sub>3</sub>	Days	Rate mmHg/day
LP-2* <sup>▲</sup>		59.3	19.3	19.7	0.36	0.44		
0.44% HNO <sub>3</sub>	25	59.7	19.9	20.0	0.33	0.48	136	0.1
0.44% HNO <sub>3</sub>	50	58.4	20.4	20.5	0.60	0.74	116	2.0
0.44% HNO <sub>3</sub>	65	57.7	20.1	20.3	1.05	1.18	48	8.5
LP-3** <sup>Δ</sup>		59.4	20.3	19.5	0.48	0.03		
2.1 ppm Fe	65	59.0	20.7	19.5	0.60	1.02	65	6.9
0.54% HNO <sub>3</sub>								
2.1 ppm Fe	65	58.6	20.4	20.1	0.40	1.52	30	13.4
0.98% HNO <sub>3</sub>								
2.1 ppm Fe	25	60.0	20.6	19.2	0.37	0.12	120	0
7.25 ppm Fe	25	59.9	20.4	20.1	0.43	0.14	120	0
51.0 ppm Fe	25	60.1	20.6	19.4	0.42	0.26	120	<0.01
49.4 ppm Cu	25	60.0	20.7	19.4	0.47	0.26	120	<0.01
2.1 ppm Fe	50	59.8	20.5	19.5	0.47	0.23	120	0
7.0 ppm Fe	50	59.5	20.7	19.6	0.56	0.28	120	0.42
23.8 ppm Fe	50	58.6	21.6	20.2	0.68	0.76	120	5.5
24.5 ppm Cu	50	58.1	22.2	20.1	0.87	0.86	120	2.8
49.9 ppm Fe	50	58.4	21.6	19.4	0.92	0.80	70	9.0
49.2 ppm Cu	50	57.5	20.3	20.2	1.40	1.03	84	5.0
2.1 ppm Fe	65	59.3	21.3	19.8	0.64	0.60	98	4.4
6.9 ppm Fe	65	58.4	21.4	19.8	0.90	0.86	78	11.0

Note:

\*--Initial composition of LP-2 (Lot # ABY87FS2C013).

\*\*--Initial composition of LP3 (Lot #1846-01).

<sup>▲</sup>-LP-2 contains 0ppm Fe, 0.7ppm Cr and 0.9ppm Ni.

<sup>Δ</sup>-LP-3 contains 2ppm Fe, 0.4ppm Cr and 0.3ppm Ni.

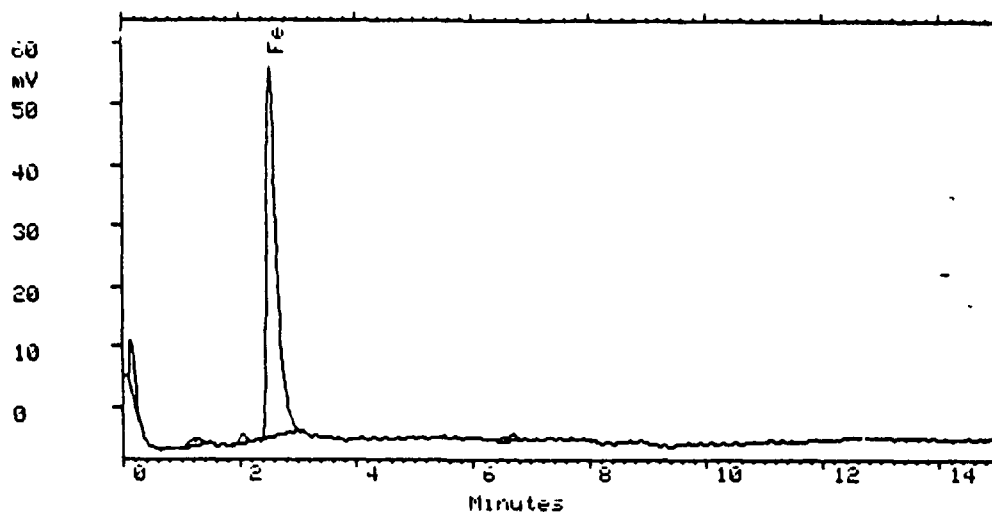
All rates are final rates in mmHg/day.

LP-2 is being reanalyzed for metals and is expected to contain higher levels than those reported by ICP/ES.

Transition Metal Method;  $\mu$ Bondapak C18  
 2mMNaOS/50mMTartaric Acid; pH=3.4w/NaOH  
 Post Col PAR Det @ 520nm; 0.5ml/min  
 UVDet; Eluant 1.0ml/minFR; first-TM's +2

FeStd: 2.8 ppm  $\text{Fe}^{+3}$  pH = 7

Chromatogram of Fe3s11



#### Conditions

Run time 15.00 min Sample rate 1.00 per sec  
 Injection volume 5  $\mu$ L Sample amount  
 Internal standard amt Scale factor  
 Mode Analysis  
 Keyboards of Remote Devices Unlocked

Peak Detect Threshold 25 Peak Width 20 sec  
 Integration Delay 0.00 min Area Reject 1

Single Point Calibration Quantitation by Area  
 Retention Time Offset 0.00 sec Force Through Zero is Disabled  
 Relative Peak Window 5% Absolute Peak Window OFF  
 Errors Reported From Integration/Quantitation:  
 Error 7 response or amount missing for all levels

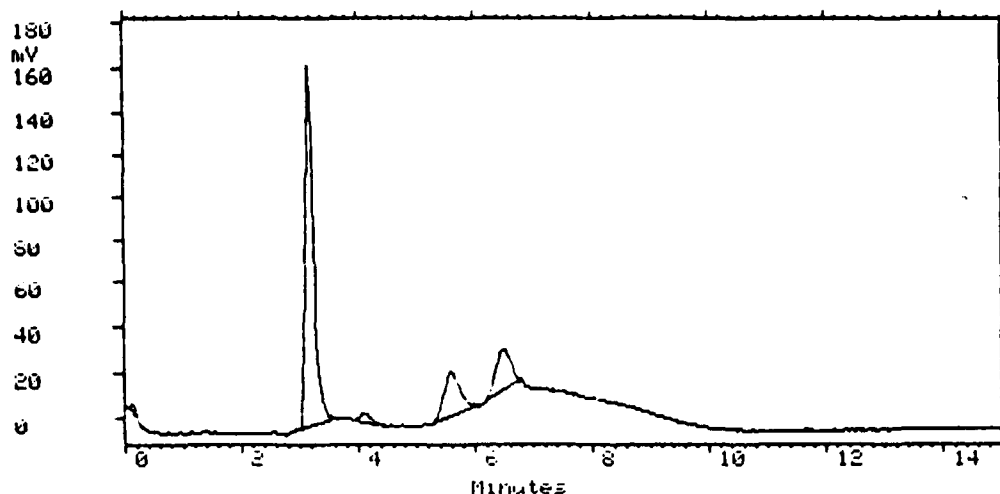
Peak Name	Ret time	Area	Height	Type	Amount	Intercept	Slope	Response
UNKNOWN	0.14	52357	9108	BB	-	-	-	-
UNKNOWN	1.29	16538	1054	BB	-	-	-	-
UNKNOWN	2.05	13502	1524	BB	-	-	-	-
Fe3	2.53	733494	61378	BB	0.000	0.000e+00	0.000e+00	7.33494e+05
Cu2	4.25	-	-	NF	-	-	-	-
Pb2	5.00	-	-	NF	-	-	-	-
Zn2	6.00	-	-	NF	-	-	-	-
UNKNOWN	6.69	12291	945	BB	-	-	-	-
Ni2	7.10	-	-	NF	-	-	-	-
Co2	9.50	-	-	NF	-	-	-	-
Cd2	10.20	-	-	NF	-	-	-	-
Fe2	11.20	-	-	NF	-	-	-	-

Figure 1. IC chromatogram of  $\text{Fe}^{+3}$  standard

Transition Metal Method;  $\mu$ Bondapak C18  
 2mMNaOS/50mMTartaric Acid; pH=3.4w/NaOH  
 Post Col PAR Det @ 520nm; 0.5ml/min  
 UVDet; Eluant 1.0ml/minFR; first-TM's +2

CuStd: 1.2 ppm pH = 7

Chromatogram of 2bCu52



#### Conditions

Run time 15.00 min Sample rate 1.00 per sec  
 Injection volume 10  $\mu$ L Sample amount  
 Internal standard amt Scale factor  
 Mode Analysis  
 Keyboards of Remote Devices Unlocked

Peak Detect Threshold 25 Peak Width 20 sec  
 Integration Delay 0.00 min Area Reject 1

Single Point Calibration Quantitation by Area  
 Retention Time Offset 0.00 sec Force Through Zero is Disabled  
 Relative Peak Window 5% Absolute Peak Window OFF

Errors Reported From Integration/Quantitation:  
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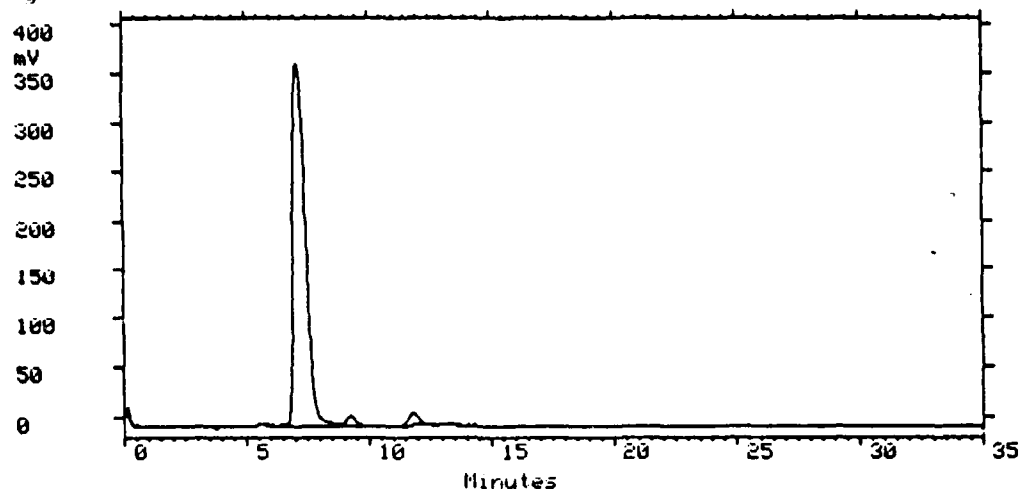
Peak Name	Ret time	Area	Height	Type	Amount	Intercept	Slope	Response
UNK	0.12	16533	3077	BB	-	-	-	-
Fe3	2.50	-	-	NF	-	-	-	-
Cu2	3.17	1607162	166407	BB	-	-	-	-
UNK	4.12	51022	3911	BB	0.000	0.000e+00	0.000e+00	5.10225e+04
UNK	5.00	-	-	NF	-	-	-	-
UNK	5.60	401257	19994	BB	-	-	-	-
UNK	6.00	-	-	NF	-	-	-	-
UNK	6.49	332402	17856	BB	-	-	-	-
Ni2	7.10	-	-	NF	-	-	-	-
Co2	9.50	-	-	NF	-	-	-	-
Cd2	10.20	-	-	NF	-	-	-	-
Fe2	11.67	3394	858	BB	0.000	0.000e+00	0.000e+00	3.39400e+03

Figure 2. IC chromatogram of  $\text{Cu}^{+2}$  standard

Transition Metal Method;  $\mu$ Bondapak C18  
 2mMNaOS/50mMTartaric Acid; pH=3.4w/NaOH  
 Post Col PAR Det @ 520nm; 0.5ml/min  
 UVDet; Eluant 1.0ml/minFR; first-TM's +2

NiStd: 1.6 ppm pH = 7

Chromatogram of Ni2st1



#### Conditions

Run time 35.00 min Sample rate 1.00 per sec  
 Injection volume 5  $\mu$ L Sample amount  
 Internal standard amt Scale factor  
 Mode Analysis  
 Keyboards of Remote Devices Unlocked

Peak Detect Threshold 25 Peak Width 20 sec  
 Integration Delay 0.00 min Area Reject 1

Single Point Calibration Quantitation by Area  
 Retention Time Offset 0.00 sec Force Through Zero is Disabled  
 Relative Peak Window 5% Absolute Peak Window OFF  
 Errors Reported From Integration/Quantitation:  
 Error 7 response or amount missing for all levels

#### Log Messages

Time Message  
 User abort.

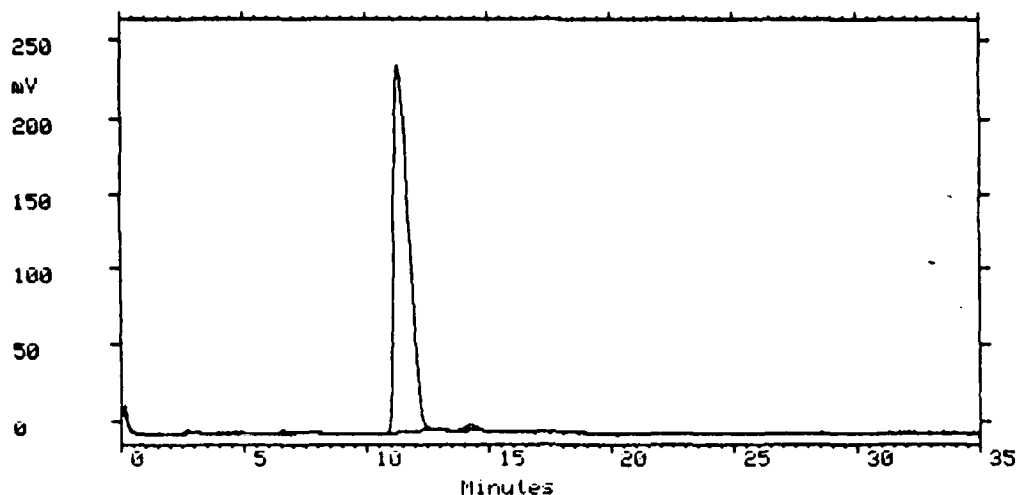
Peak Name	Ret time	Area	Height	Type	Amount	Intercept	Slope	Response
UNKNOWN	0.12	52783	8638	BB	-	-	-	-
UNKNOWN	0.49	12199	1852	BB	-	-	-	-
Fe3	2.30	-	-	NF	-	-	-	-
UNKNOWN	3.66	3412	508	BB	-	-	-	-
UNK	4.70	-	-	NF	-	-	-	-
UNK	6.70	-	-	NF	-	-	-	-
Ni2	7.11	12866378	369832	BV	-	-	-	-
UNKNOWN	9.26	282911	9303	VB	-	-	-	-
UNK	11.79	369482	12308	BB	0.000	0.000e+00	0.000e+00	3.69482e+05

Figure 3. IC chromatogram of Ni<sup>2+</sup> standard

Transition Metal Method;  $\mu$ Bondapak C18  
 2mMNaOS/50mMTartaric Acid; pH=3.4w/NaOH  
 Post Col PAR Det @ 520nm; 0.5ml/min  
 UVDet; Eluant 1.0ml/minFR; first-TM's +2

FeStd: 3.4 ppm  $\text{Fe}^{+2}$  pH = 7

Chromatogram of Fe2st1



#### Conditions

Run time 35.00 min Sample rate 1.00 per sec  
 Injection volume 5  $\mu$ L Sample amount  
 Internal standard amt Scale factor  
 Mode Analysis  
 Keyboards of Remote Devices Unlocked

Peak Detect Threshold 25 Peak Width 20 sec  
 Integration Delay 0.00 min Area Reject 1

Single Point Calibration Quantitation by Area  
 Retention Time Offset 0.00 sec Force Through Zero is Disabled  
 Relative Peak Window 5% Absolute Peak Window OFF

Errors Reported From Integration/Quantitation:

Error 7 response or amount missing for all levels

Peak Name	Ret time	Area	Height	Type	Amount	Intercept	Slope	Response
UNKNOWN	0.12	44558	7573	BB	-	-	-	-
Fe3	2.30	-	-	NF	-	-	-	-
UNKNOWN	2.71	32446	2570	BB	-	-	-	-
UNKNOWN	3.13	11028	997	BB	-	-	-	-
UNKNOWN	3.73	12846	552	BB	-	-	-	-
UNKNOWN	4.52	5696	842	BV	-	-	-	-
UNKNOWN	4.73	7102	1577	VB	0.000	0.000e+00	0.000e+00	7.10153e+03
UNKNOWN	4.93	7503	822	BB	-	-	-	-
Pb2	6.57	38483	2320	BB	0.000	0.000e+00	0.000e+00	3.84830e+04
UNKNOWN	7.33	7276	553	BB	-	-	-	-
UNKNOWN	9.93	2574	462	BB	-	-	-	-
Fe2	11.34	10255250	242745	BB	-	-	-	-

Figure 4. IC chromatogram of  $\text{Fe}^{+2}$  standard

Pump: Waters Model 510  
Column: Waters 15 cm NOVA PAK C18  
Eluent: 5 mM PIC A in 10% CH<sub>3</sub>CN  
Flow Rate: 1.0 mL/min  
Injector: Waters Model U6K  
Inj. Vol: 50  $\mu$ L  
Detector: Waters UV/VIS Model 481 @ 365 nm

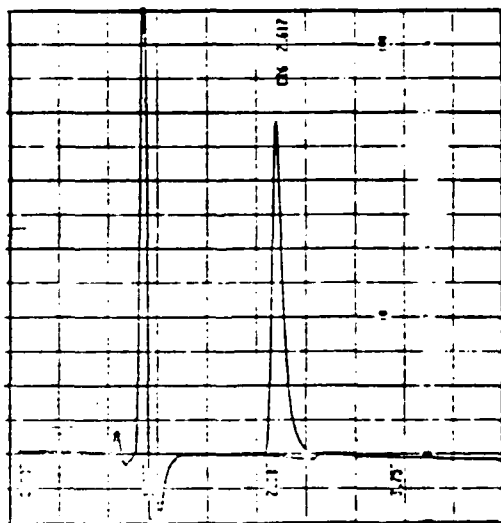


Figure 5. IC chromatogram of Cr<sup>+6</sup> standard



HWCKK1

3-Nov-88

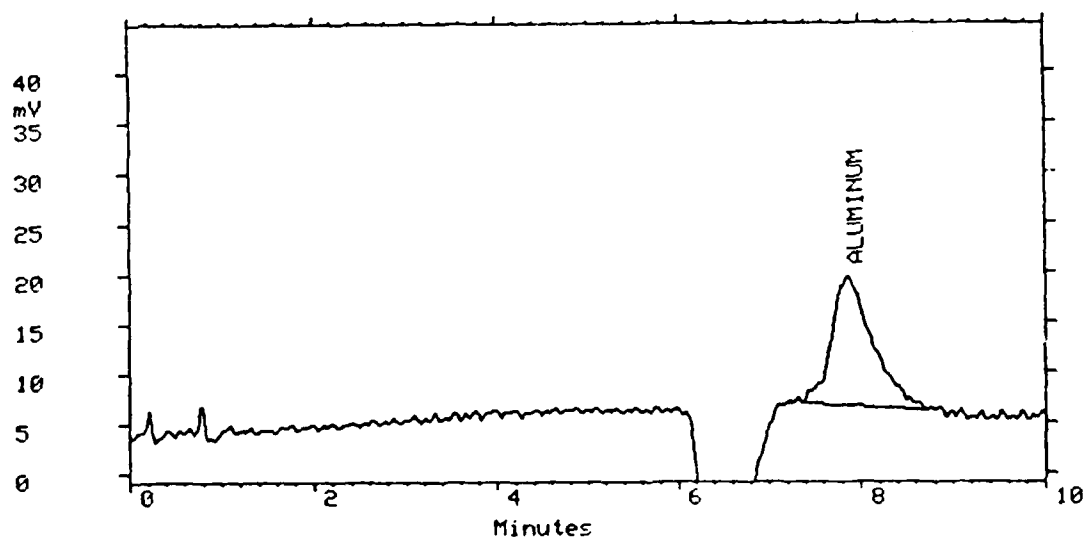
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Acquisition method	FIA	Quantitation method	FIA
Units	PPB	System number	2
Channel	1	Manual injector	
Injection	1	Total injections	1
Run time	10.00 min	Sample rate	1.00 per sec
Injection volume	100 $\mu$ L	Mode	Analysis
Acquisition version	6.2	Quantitation version	6.2

UV 570 NM

Chromatogram of HWCKK1



Peak Name	Ret time	Area	Height	Type	Amount	Intercept	Slope	Response
ALUMINUM	7.87	395978	12886	BB	102.371	0.000e+00	3.868e+03	3.95978e+05

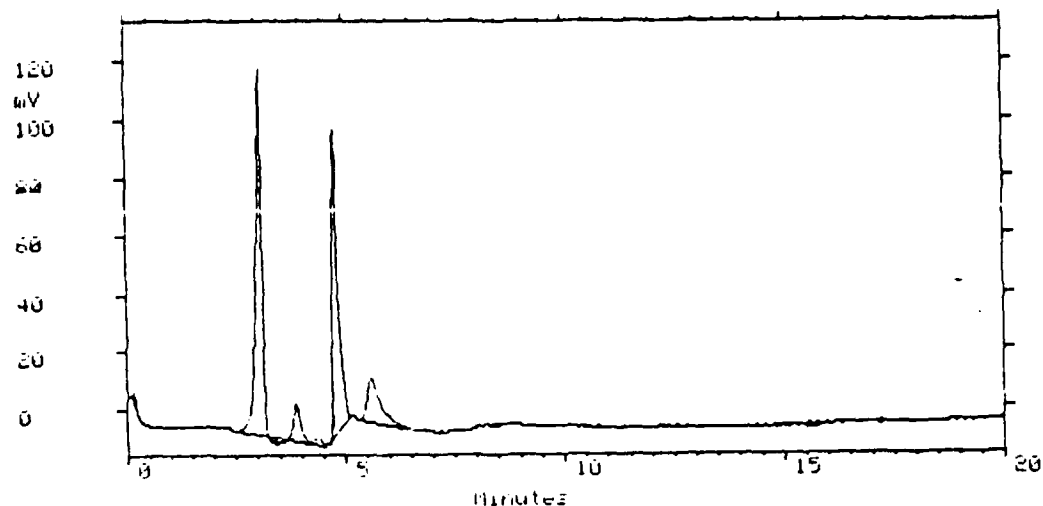
CORRECTED = 102.89 ppb Al

Figure 6. IC chromatogram of  $\text{Al}^{13}$  standard

Transition Metal Method;  $\mu$ Bondapak C18  
 2mMNaOS/50mMTartaric Acid; pH=3.4w/NaOH  
 Post Col PAR Det @ 520nm; 0.5ml/min  
 UVDet; Eluant 1.0ml/minFR; first-TM's +2

LP 1846-03-11 3.006g digested in 25ml pH=1

Chromatogram of 4601b



#### Conditions

Run time 20.00 min Sample rate 1.00 per sec  
 Injection volume 100  $\mu$ L Sample amount  
 Internal standard amt Scale factor  
 Mode Analysis  
 Keyboards of Remote Devices Unlocked

Peak Detect Threshold 25 Peak Width 20 sec  
 Integration Delay 0.00 min Area Reject 1

Single Point Calibration Quantitation by Area  
 Retention Time Offset 0.00 sec Force Through Zero is Disabled  
 Relative Peak Window 5% Absolute Peak Window OFF  
 Errors Reported From Integration/Quantitation:

Error 7 response or amount missing for all levels

Peak Name	Ret time	Area	Height	Type	Amount	Intercept	Slope	Response
UNKNOWN	0.12	15127	2797	BB	-	-	-	-
UNK	2.50	-	-	NF	-	-	-	-
Fe3	3.08	183264	125510	BV	0.70	-	-	-
UNK	3.87	39962	13516	VV	-	-	-	-
UNK	4.38	4150	2173	VB	0.000	0.000e+00	0.000e+00	-
Cd2	4.77	44195	107570	BB	0.000	0.000e+00	0.000e+00	-
UNKNOWN	5.60	41214	15562	bb	-	-	-	-
Zn2	6.00	-	-	NF	-	-	-	-
Ni2	7.10	-	-	NF	-	-	-	-
Co2	9.50	-	-	NF	-	-	-	-
Cd2	10.20	-	-	NF	-	-	-	-
Fe2	11.20	-	-	NF	-	-	-	-

Figure 7. IC chromatogram of LP 1846

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3. Griff, S. P.; Doyle, G.; Turngren, E.; Seals, W. O.; and Bracuti, A., "Development of Analytical Techniques for Liquid Propellant Surveillance," JANNAF Propellant Development and Characterization Subcommittee Meeting, Laurel, MD, November 1989, CPIA publication, Contractor Report ARAED-CR-89016, February, 1990.

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